Determination of Several Minor Elements in Aluminium Alloys by Proton Activation Analysis using a New Internal Standard Method

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In a previous paper[1)], we have been proposed a new internal standard method for activation analysis. The method has great characteristics in that a suitable element present originally in the sample is used as an internal standard, and the comparative standard is prepared by applying the standard addition method to the duplicated sample. The outline of the method may be written as follows. When a sample under examination contains W_a g of a trace element A to be determined together with an element B which is usable as an internal standard, and when the comparative standard is prepared by adding W_a* g of accurately known small amount of the element A to the duplicated sample, W_a can be determined easily by using the following equation even if the sample and the comparative standard are irradiated separately by particles with different flux.

$$W_a = W_a*/ [(A_R*/A_R) - 1]$$

where A_R and A_R* are counting ratios of gamma-rays emitted by two radionuclides produced from the element A and B in the sample and the comparative standard, respectively. In principle, this method can be applied to multielements determination in a wide variety of materials by various activation methods.

Although the usefulness of the method was demonstrated previously through the determination of several trace elements in pepperbush by means of photon activation. In the present study, the method was further examined through the determination of Ti, Cr, Fe, Cu, Ga and Zr in commercial aluminium alloys by using proton inuced reactions.

Aluminium Association No. 7079 alloy was used as a sample. Zinc was used as an internal standard because ^{67}Ga(78.3 h) produced by the $^{67}Zn(p,n)$ reaction has the suitable half-life for measurement and has characteristic gamma-rays in the appropriate energy range. In order to fit the chemical composition of the sample and that of the comparative standard, preparation of the sample was carried out according to the procedure described by Mitchell et al.[2)] After about 0.1 g of the sample slices was dissolved in 6N HCl, the solution was processed by using tetraethylsilicate. The comparative standard containing known amounts of Ti, Cr, Fe, Cu, Ga and Zr was also prepared by the same procedure as above. The resulting powder of sample or comparative standard was then pressed into a smooth-surface pellet, and wrapped in a pure aluminium foil for irradiation. After the sample and the comparative standard pellets prepared as above were set in a rotating-target assembly, they were irradiated with 4 μA beam of 13 MeV protons for 2 hours. Gamma-ray spectrum of each pellet was measured by using a high resolution Ge(Li) detector coupled with a multichannel pulse height analyser.

The gamma-ray spectrum of AA-7079 measured 2.5 days after the end of irradiation is shown in Fig. 1. Several radionuclides shown in the figure were identified. Quantitative determinations of Ti, Cr, Fe, Cu, Ga and Zr were carried out as follows by measuring the gamma-ray spectra of the irradiated sample and the comparative standard. Since titanium gives ^{48}V(15.976 d) by the $^{48}Ti(p,n)$ reaction, the A_R and A_R* were obtained as the ratios of the net photopeak countings due to 984 keV line (^{48}V) to those due to 185 keV line (^{67}Ga). Chromium produces ^{52}Mn(5.59 d) by the $^{52}Cr(p,n)$ reaction, so that the A_R and A_R* were obtained as the ratios of the net photopeak countings due to 744 keV line (^{52}Mn) to those due to 185 keV line (^{67}Ga). Iron also results in ^{56}Co (78.3 d) by the $^{56}Fe(p,n)$ reaction. Hence, the A_R and A_R* were obtained as the ratios of the net photopeak countings due to 847 keV line (^{56}Co) to those due to 185 keV line (^{67}Ga). Copper also produces ^{65}Zn by the $^{65}Cu(p,n)$ reaction. The A_R and A_R* were therefore obtained as the ratios of the net photopeak countings due to 1116 keV line (^{65}Zn) to those due to 185 keV line (^{67}Ga). Gallium also results in ^{69}Ge(39.0 h) by the $^{69}Ga(p,n)$ reaction. Hence, the A_R and A_R* were obtained as the ratios of the net photopeak countings due to 574 keV line (^{69}Ge) to those due to 185 keV line (^{67}Ga). On the other hand, zirconium also leads ^{90}Nb(14.6 h) by the $^{90}Zr(p,n)$ reaction. Accordingly, the A_R and A_R* were obtained as the ratios of the net photopeak countings due to 141 keV line (^{90}Nb) to those due to 185 keV line (^{67}Ga).

The results obtained by three replicate analyses are shown in Table 1 together with the values given by the other analytical methods. The values obtained for each element to be determined were found to be reproducible within the statistical limit, and were in good agreement with the reference values. As demonstrated above, the present internal standard method was also proved to be an effective method for proton activation analysis.

References

1) Yagi M. and Masumoto K., J. Radioanal. Nucl. Chem. 83 (1984) 313.
2) Mitchell J.W., Blitzer L. D., Kometani T. Y., Gills T. and Clark L. Jr., J. Radioanal. Chem. 39 (1979) 335.

Table 1. Concentrations of minor elements in commercial aluminium alloy AA 7079 (mass %).

Element	This work	Average	Other methods
Ti	0.0364 0.0369	0.0367±0.0003	0.04[a] 0.040[b] 0.0355[c] 0.03[d]
Cr	0.127 0.127 0.126	0.127 ±0.001	0.13[a] 0.133[b] 0.124[c] 0.14[d]
Fe	0.345 0.337 0.331	0.338 ±0.007	0.36[a] 0.35[b] 0.26[d]
Cu	0.713 0.708 0.669	0.697 ±0.024	0.65[a] 0.68[c] 0.87[d]
Ga	0.0095 0.0087 0.0099	0.0093±0.0006	0.03[a] 0.014[c]
Zr	0.0492 0.0510 0.0565	0.0523±0.0038	ND[a] 0.052[b] 0.048[c] 0.01[d]

a Emission spectrometry,
b Proton activation analysis (Comparative method),
c Photon activation analysis,
d Presented value by Alcan.

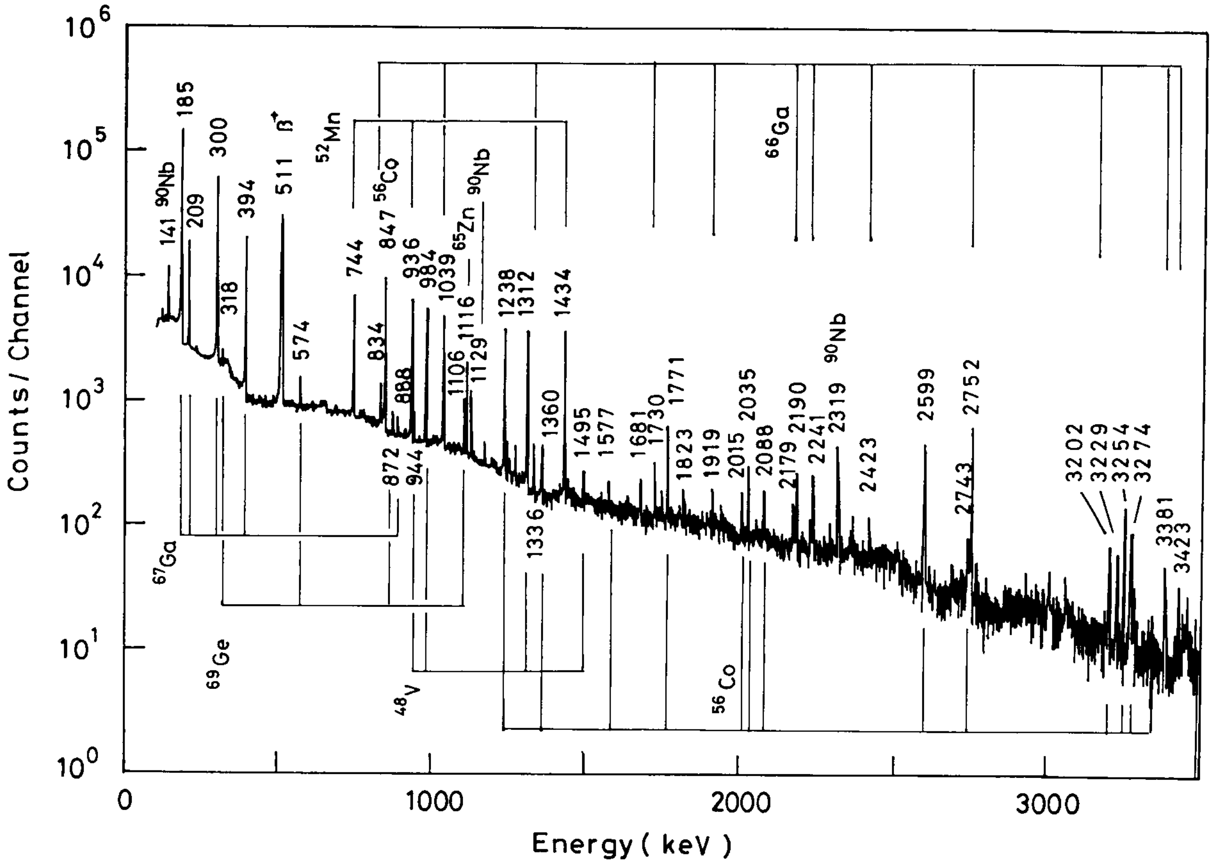

Fig. 1. Gamma-ray spectrum of AA-7079 measured 2.5 days after the end of irradation.